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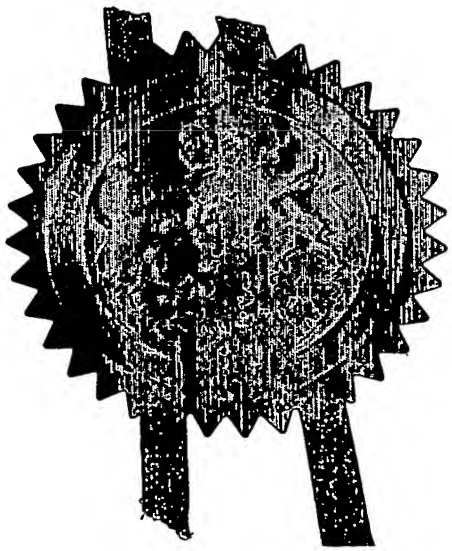

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Dated 26 August 2004

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1	Your reference	UQI 51109/GB
2	Patent application number *(The Patent Office will fill in this part)*	0318663.2
3	Full name, address and postcode of the or of each applicant (underline all surnames)	Imperial Chemical Industries plc 20 Manchester Square London W1U 3AN
	Patents ADP Number *(if you know it)*	0000093506 00000935006
	If the applicant is a corporate body, give the country/state of its incorporation	United Kingdom
4	Title of the invention	Rolling Formulation
5	Name of Your Agent *(if you have one)*	RALPH, Julie Diane
	"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*	Uniqema Intellectual Property Dept Wilton Centre, Wilton Redcar Cleveland England, TS10 4RF
	Patents ADP Number *(if you know it)*	07591688900 2

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7	If this application is divided or otherwise derived from an earlier UK application, give the number and filing date of the earlier application		Number of earlier application	Date of Filing *(day / month / year)*
8	Is a statement of inventorship and of right to grant of a patent required in support of this request?	Yes		

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Patents Form 1/77

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Continuation sheets of this form	-
Description	7
Claim(s)	1
Abstract	0
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Translations of priority documents	
Statement of Invention and right to grant of a patent *(Patents Form 7/77)*	
Request for Preliminary Examination and search *(Patents Form 9/77)*	
Request for Substantive Examination *(Patents Form 10/77)*	
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11 I/We request the grant of a patent on the basis of this application

Signature Julie Ralph Date 8 Aug 2003

12 Name and daytime telephone number of person to contact in the United Kingdom — Julie Diane RALPH 01642 435334

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Rolling formulation

The present invention relates to a water-soluble cold steel rolling oil composition and an oil-in water emulsion comprising water and the water-soluble cold steel rolling oil.

Cold steel rolling is a process which can be applied to steel sheets of about 0.5 to 1cm thick, which have been produced by a hot rolling process from thick (e.g. 20cm) slabs of hot steel, to further reduce the sheets to much lighter gauges of typically 0.15 to 0.35mm. The cold steel rolling process is a balance of load applied through each series of rolls, front and back tension applied to the steel sheet and rolling speed though the rolls to achieve a steel sheet product of saleable quality. The rolled steel is highly stressed during the cold rolling process and must be then relaxed to ensure that the steel is in the correct crystalline form for its designated application. Relaxation is achieved by an annealing process.

The surface quality of cold rolled annealed steel sheets can be marred by the presence of black staining and surface carbon residues. Such steel sheet surface quality may not be suitable for use in automotive application areas where there is a designated maximum level of allowable surface carbon residues of 7mg/m^2 for mill clean sheet. However this sheet surface quality requirement appears to be in direct conflict with the pressures placed on mill owners to continually seek to run their mills at faster speeds to improve output, to reduce power consumption of the mill and to roll different and harder alloys of steel.

This had led to rolling oil formulators formulating rolling oils that can "plate out" a greater amount of oil to try to address the pressures placed on the mill owners. However the increase in "plated out" oil needs to be readily removable during the annealing process, typically at 600 to 700 degC for batch annealing processes, to meet the steel sheet surface quality requirement.

One mechanism that has been suggested for the generation of the black staining patches and surface carbon residues is as follows. After the cold rolling process the steel sheet is primarily contaminated with iron fines and formulating oil residues. Typically the formulating oil residues can contain tramp oils, soaps and products of the rolling process. Deformation and frictional heat generated during the cold rolling process, of 100deg C or higher, can persist in the rolled coil for a few hours. Under these conditions oxidation and polymerisation of some of the components in the

formulating oil residues may lead to the formation of less volatile macromolecular substances. These reactions may be accelerated in the presence of the iron fines acting as a catalyst. It is thought that these less volatile macromolecular substances are carbonised during the annealing process, which results in the generation of the black staining patches and surface carbon residues.

Therefore the staining patches and surface carbon residues seem to be a function of the combination of the heat generated during the cold rolling process, contaminants in the cold rolling process and the composition of the rolling oil formulation.

Surprisingly it has been found that the use of a specific partial polyol ester in the rolling oil formulation meets the "plate –out" requirements of the mill owners and the requirements for steel sheet surface quality without any corresponding detriment to any of the other water-soluble rolling oil composition requirements, in particular lubricity.

According to the present invention, a water-soluble rolling oil composition for use in cold steel rolling applications comprises 15 to 70 % by weight of a partial polyol ester having a hydroxyl value of between 20 and 60 mg KOH/g and a level of polyunsaturation of between 0.01 to 8% by weight.

In the context of the invention polyunsaturation is defined so as to exclude monounsaturation.

The partial polyol ester may be derived from the reaction of at least one polyhydric alcohol with at least one monocarboxylic acid. Alternatively it may be derived from the blending together of at least two partial polyol esters or the blending together of a combination of at least one partial polyol ester and at least one fully esterified ester.

The polyhydric alcohol may be a diol, triol, tetraol and/or related dimers and trimers. Examples are neopentyl glycol, glycerol, trimethylolethane, trimethylolpropane, trimethylolbutane, pentaerythritol, dipentaerthyritol and tripentaerythritol. Preferably the polyhydric alcohol is chosen from neopentyl glycol, trimethylolpropane and pentaerythritol, especially trimethylolpropane.

The polyunsaturation in the partial polyol ester is derived from the at least one monocarboxylic acid. The at least one monocarboxylic acid may be, for example, one

or more polyunsaturated monocarboxylic acids or a mixture of polyunsaturated and non-polyunsaturated monocarboxylic acids.

The polyunsaturated or non-polyunsaturated monocarboxylic acid may have some monounsaturated carbon –carbon bonds and may have a linear and/or branched chain. It may be aliphatic or aromatic. Preferably the polyunsaturated or non-polyunsaturated monocarboxylic fatty acid has 8-24 carbon atoms, more preferably 10-20 carbon atoms. Examples of suitable polyunsaturated monocarboxylic fatty acids include those derived from coconut oil, topped coconut oil (coconut oil which has been further processed to remove the C8 and C10 saturated fatty acid components), palm kernel oil and mixtures thereof. Examples of suitable non-polyunsaturated monocarboxylic fatty acids include capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, isostearic acid, hydrogenated C18 monomeric acid, arachidic acid, behenic acid and lignoceric acid and mixtures thereof. Hydrogenated C18 monomeric acid is the C18 monomer by-product resulting from the dimerisation of oleic acid, which is then subsequently hydrogenated.

A specifically preferred partial polyol ester is derived from reaction of trimethylolpropane with a mixture of coconut oil or topped coconut oil and hydrogenated C18 monomeric acid. The blend ratio of coconut oil or topped coconut oil to hydrogenated C18 monomeric acid in the partial ester composition preferably ranges from 80 to 20 weight percent to 20 to 80 weight percent, more preferably from 60 to 40 weight percent to 40 to 60 weight percent.

The partial polyol ester according to the invention is present at levels between 15 and 70% by weight, more preferably between 20 and 50%, even more preferably between 20 and 40% in the water-soluble rolling oil composition.

The partial polyol ester has a hydroxyl value of between 20 and 50 mg KOH/g, preferably between 25 and 40 mg KOH/g.

The partial polyol ester has a level of polyunsaturation of between 0.01 to 8% by weight, preferably between 0.1 to 5% by weight, more preferably between 0.1 to 3% by weight.

The kinematic viscosity at 40 degrees C of the partial polyol ester is between 40 and 80 mm^2/s, preferably between 50 and 60 mm^2/s and the pour point is between -10 and +10 degrees C.

The water-soluble rolling oil composition may further comprise other esters in the form of natural oils/fats, for example coconut oil, palm oil, tallow, sunflower oil, and mineral oil, in particular paraffinic mineral oil.

The water-soluble rolling oil composition may further comprise a range of additives. Preferred additives include antioxidants, for example sterically hindered phenols, aminic antioxidants and butylated hydroxy toluene, antiwear/extreme pressure additives, for example dilauryl phosphate, tri (2-ethylhexyl) phosphate, tricresylphosphate , zinc dialkyl (or diaryl) dithiophosphate, phospho-sulphurised fatty oils, zinc dialkyl dithiocarbamate, mercaptobenzothiazole sulphurized fatty oils and alkyl or aryl polysulphides and emulsifiers, for example nonylphenol ethoxylates, fatty alcohol ethoxylates, polyethylene glycol esters, synthetic alcohol ethoxylates and polymeric surfactants.

An example of a typical water-soluble rolling oil composition according to the invention is:

Partial polyol ester	20-70 wt %
Natural oil/fat	0-20 wt %
Mineral oil	20-50 wt%
Antioxidant	0.5-1wt%
Anti wear/Extreme Pressure additive(s)	2-5wt%
Emulsifier(s)	5-7wt%

According to a further embodiment of the present invention use of a water-soluble rolling oil composition which comprises 15 to 70 % by weight of a partial polyol ester having a hydroxyl value of between 20 and 50 mg KOH/g and a level of polyunsaturation of between 0.01 to 8% by weight in cold steel rolling applications.

According to a further embodiment of the present invention an oil in water emulsion comprises water and from 1-4% by weight of a water-soluble rolling oil composition for use in cold steel rolling applications which comprises 15 to 70 % by weight of a partial polyol ester having a hydroxyl value of between 20 and 50 mg KOH/g and a level of polyunsaturation of between 0.01 to 8% by weight.

Preferably the oil in water emulsion comprises from 2-3% by weight of the water-soluble rolling oil composition.

According to a further embodiment of the present invention use of an oil in water emulsion comprises water and from 1-4% by weight of a water-soluble rolling oil composition which comprises 15 to 70 % by weight of a partial polyol ester having a hydroxyl value of between 20 and 60 mg KOH/g and a level of polyunsaturation of between 0.01 to 8% by weight in cold steel rolling applications.

The invention will now be described further by way of example only with reference to the following Examples and drawings, in which: Figure One is a pictorial representation of the results obtained in Example One and Figure Two is a graphical representation of the results obtained in Example Three.

Example One

An in-house test was developed to mimic the effect of high temperature on the water-soluble rolling oil composition. A neat ester solution was applied as a thin film to a clean, dry, aluminium tray. The tray was heated on a hotplate, in a well ventilated area, to about 300 degree C and held at this temperature for 10 minutes.

Figure One illustrates two aluminium trays after the heating process for sample A, which is a partial polyol ester according to the invention, and Sample B, which is a comparative ester which is used commercially in water-soluble rolling oil compositions in cold steel rolling. Sample A is a partial polyol ester derived from the reaction of trimethylolpropane with topped coconut oil and hydrogenated C18 monomeric acid in a ratio of 60:40 weight percent. Sample A has a hydroxyl value of 36 mgKOH/g and a polyunsaturation level of 2.4%. Comparative sample B is PRIOLUBE 1427 available ex Uniqema and is an ester of trimethylolpropane with oleic acid having a hydroxyl value of 8mgKOH/g and a polyunsaturation level of 14%.

The aluminium tray with Sample B shows evidence of dark coloured residues, which are hardly evident for the tray with Sample A.

Example Two

Experiments were undertaken to measure the clean burning properties of Samples A and B and a further Sample according to the invention, Sample C.
20 to 25mg of each Sample was heated, in a 150µl alumina cup, from 40 to 600°C at a rate of 10 °C /min under a nitrogen flow of 100ml/min. The temperature was measured for a variety of levels of percentage evaporation loss of sample. The results are shown in Table One.

Table One

% sample remaining	75%	50%	30%	20%	10%	1%
Sample A temperatures in °C	377	411	426	432	439	452
Sample C temperatures in °C	382	417	431	437	444	457
Comparative Sample B temperature in °C	417	437	448	454	462	Not Measured

Sample C is a partial polyol ester derived from the reaction of trimethylolpropane with topped coconut oil and hydrogenated C18 monomeric acid in a ratio of 40:60 weight percent. It has a hydroxyl value of 40 mgKOH/g and a polyunsaturation level of 1.6%. The results in the Table demonstrate that the use of partial polyol esters in accordance with the invention have improved clean burning properties with respect to the comparative ester.

Example Three

The coefficient of friction of Samples A and B was determined at a temperature of 40°C in a Mini-Traction Machine, manufactured and supplied by PCS Instruments in the UK, using a pin-on-disc method. The disc is a smooth steel disc with a roughness parameter of 10nm. The load applied was 5N and the speed of rotation was varied from 0.002 - 4m/s to ensure that the system operated under both boundary and mixed lubrication regimes. The results are illustrated in Table Two and Figure Two.

Table Two

Speed in m/s	Coefficient of friction Sample A	Coefficient of friction Sample B
4.00	0.046	0.071
3.00	0.048	0.078
2.50	0.049	0.082
2.00	0.050	0.085
1.75	0.051	0.088
1.50	0.053	0.090
1.25	0.056	0.093
1.00	0.060	0.097
0.80	0.065	0.101
0.60	0.073	0.104
0.40	0.083	0.108
0.30	0.089	0.110
0.20	0.097	0.113
0.15	0.101	0.114
0.10	0.105	0.115
0.08	0.108	0.116
0.06	0.110	0.117
0.04	0.113	0.117
0.03	0.114	0.117
0.02	0.116	0.117

The results in Table Two and Figure Two demonstrate that the use of a partial polyol ester in accordance with the invention has improved lubricity with respect to the comparative ester. The contribution of the partial polyol ester in the water-soluble rolling oil composition to improved lubricity corresponds to reduced power consumption on a mill and improved surface finish of the rolled metal.

Claims

1. A water-soluble rolling oil composition for use in cold steel rolling applications comprising 15 to 70 % by weight of a partial polyol ester having a hydroxyl value of between 20 and 60 mg KOH/g and a level of polyunsaturation of between 0.01 to 8% by weight.

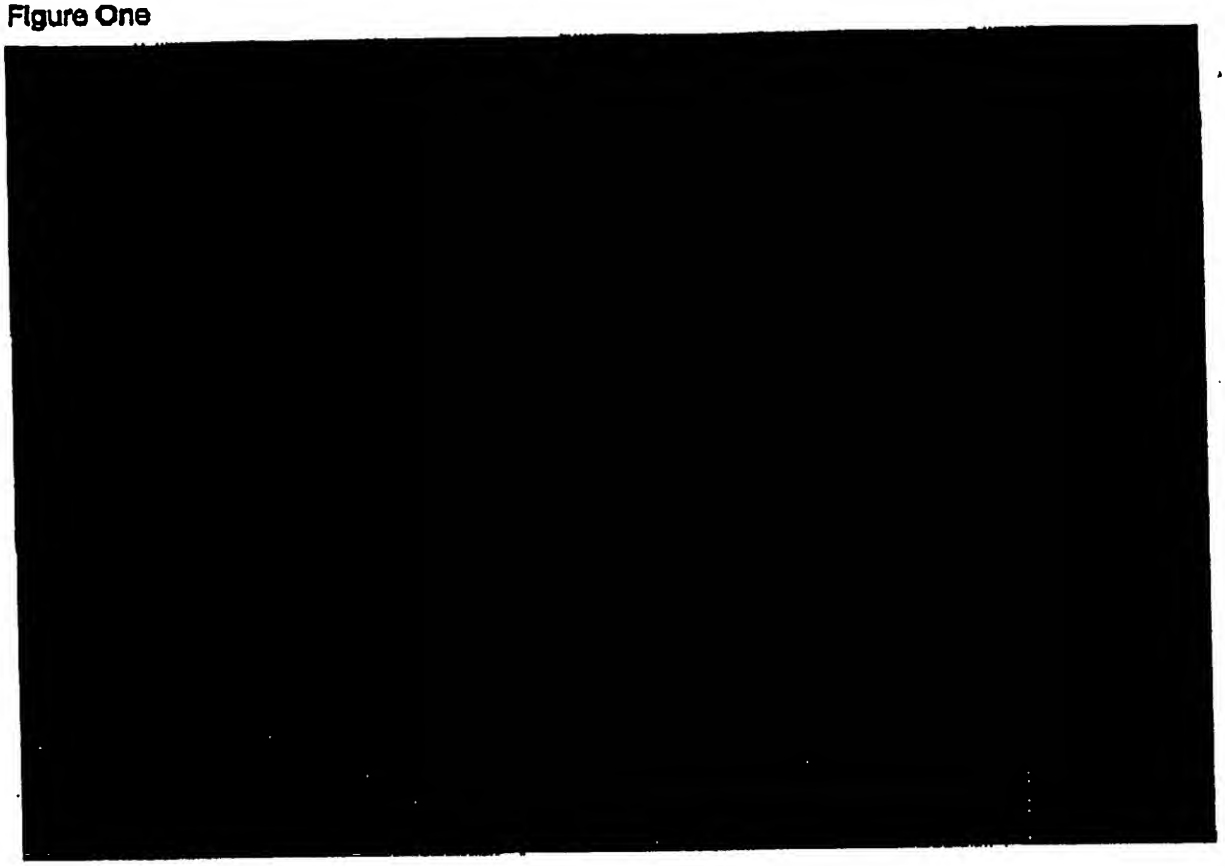
2. A water-soluble rolling oil composition as claimed in claim 1 further comprising other esters in the form of natural oils/fats and mineral oil.

3. A water-soluble rolling oil composition as claimed in claim 2 further comprising a range of additives.

4. Use of a water-soluble rolling oil composition which comprises 15 to 70 % by weight of a partial polyol ester having a hydroxyl value of between 20 and 60 mg KOH/g and a level of polyunsaturation of between 0.01 to 8% by weight in cold steel rolling applications.

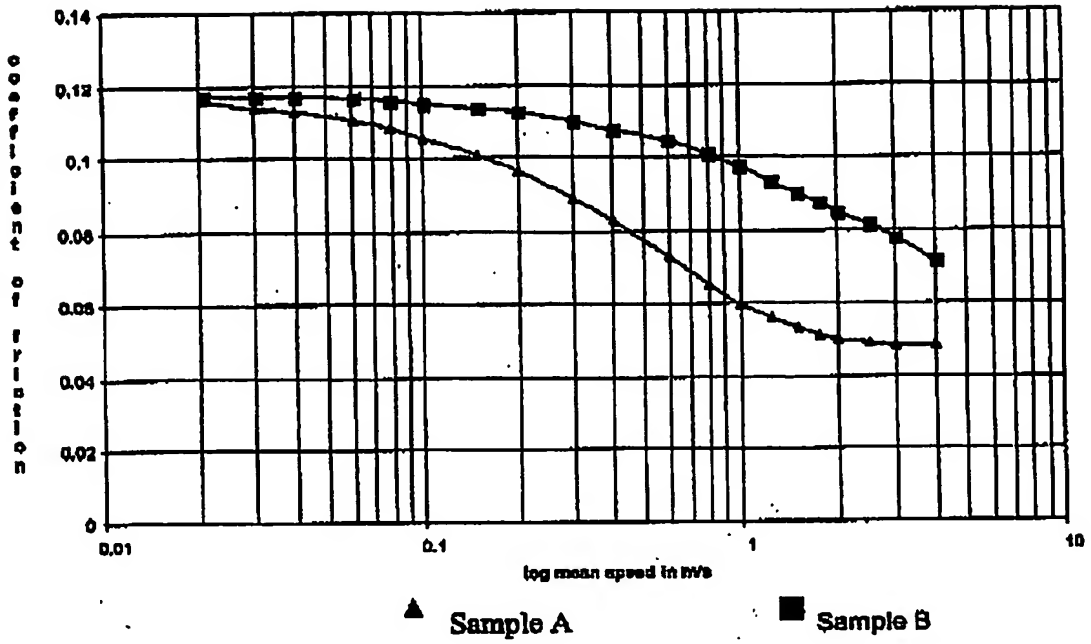
5. An oil in water emulsion comprising water and from 1-4% by weight of a water-soluble rolling oil composition for use in cold steel rolling applications which comprises 15 to 70 % by weight of a partial polyol ester having a hydroxyl value of between 20 and 60 mg KOH/g and a level of polyunsaturation of between 0.01 to 8% by weight.

Figure One



SAMPLE B SAMPLE A

Figure Two


0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0.01
0.1
1
10
log mean speed in m/s
Sample A
Sample B